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Effect of Alternating Freezing & Thawing Of Cement Mortar

Civil Engineering
B. S.
1905

EFFECT
OF
ALTERNATE FREEZING AND THAWING
OF
CEMENT MORTAR

BY

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THESIS

FOR

DEGREE OF BACHELOR OF SCIENCE

IN

CIVIL ENGINEERING

COLLEGE OF ENGINEERING

UNIVERSITY OF ILLINOIS

PRESENTED JUNE 1905

UNIVERSITY OF ILLINOIS

May 15, 1905

THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

FRANK WILLIAM HILLMAN

ENTITLED EFFECT OF ALTERNATE FREEZING AND THAWING OF CEMENT MORTAR

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE DEGREE

OF Bachelor of Science in Civil Engineering

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HEAD OF DEPARTMENT OF Civil Engineering

Effect of Alternate Freezing and Thawing of Cement Mortar.

During the summer of 1904 the writer heard an argument as to whether or not cement ~~mortar~~ was injured by freezing. The argument seemed to indicate that but little was known about this matter. Thinking that some valuable information might be obtained, the writer decided to make an investigation along this line for his thesis.

It was thought that the damage by freezing might be prevented if the activity of the cement were increased. There are a number of substances which are reported to materially effect the activity of cement. For example, Baker's Masonry Construction states that carbonate of soda mixed with cement will sometimes quicken the set. An item was read to the class to the effect that a mixture of potash lye and salt used with the cement made it set hard in

one minute. An extended search by the writer failed to find this item, and therefore no more definite information can be given. It was decided to experiment with these materials, and see if the effect of freezing could be counteracted by increasing the activity of the cement. In addition to using materials that would increase the cement's activity, it was decided to use also a salt solution, as salt has been much used in practice to retard or prevent freezing.

Nothing was known as to the proper proportions of soda and cement to use. The article from which the information concerning the use of the potash lye and salt mixture obtained stated that the lye and salt had been used one part of salt to four of lye. However it could not be learned why or how this proportion was determined. This was the proportion used in experiments refered to later. The usual rule for the proportion of salt to be used is "1 pound for 18 gallons of water when the temperature is 32° F. and add 1 ounce for each degree below freezing"; but in these experiments the following rule was used which gives substantially the same results as above: "add 1 per cent of salt for each Fahrenheit degree below freezing".

The writer asked at a drug store for carbonate of soda: but received bicarbonate. However the error was not discovered until after some tests had been made with the bicarbonate. As the bicarbonate when mixed with the cement hastened the activity, it was used throughout the experiments. Lewis' potash lye was employed. The salt used with the lye and also separately was common coarse salt.

Determination of Proportions.

To determine what proportion of soda and of potash lye and salt should be used, several preliminary experiments were made. These experiments consisted in making pats of mortar from cement and soda in varying proportions, and from the potash lye and salt mixture and cement also in varying proportions. The pats of mortar were allowed to set, and the time that the different mortars required to attain initial and final set was noted. To determine when the mortar had initial or final set Gillmor's method was used. In this method two differently weighted brass needles are employed. The cement has attained it's initial set when the mortar will support the light needle, and it's final set when it will support the heavy needle. The

time is counted from the instant the water is added to the instant the needles are just supported by the mortar.

The amount of water used in these experiments was that quantity which would make a plastic mortar. This quantity was used because it is almost the universal custom at the present time to use wet mortar or wet concrete. The amount was determined by adding water until the mortar would just slip from the trowel and work as indicated by the Boulogne test for plastic mortar.

Tests with Bicarbonate of Soda. The first experiments to determine the proper proportion of soda were made with Owl Portland cement. Twenty-five per cent of water was used, this having been determined as the proper amount to use to make a plastic mortar. With 25 per cent of soda the cement set before it could be made into pats. It is very evident that such a proportion as this is impracticable, but it was used merely to get a start. The amount of soda was then decreased successively to 12½ o/o, 8 o/o, and 4 o/o, the result being that the time required for the cement to set hard was respectively 12, 21, and 52 minutes. The cement without any soda required $1^h 45^m$ to set hard. In a previous experiment this same cement had taken $2^h 35^m$ to obtain an initial set. Why there

should be such a difference can not be explained. It is known that the temperature of the surrounding atmosphere will cause a variance in the time required for cement to set. The room at the time the first mentioned tests were made was very warm; but it does not seem reasonable to give this as the cause of the difference.

The fact of so wide a divergence in the time of set without any soda, cast a suspicion upon the results obtained with soda; and therefore it was decided to repeat the series of tests. However it was necessary to decide quickly what proportion of soda should be used so as to start the freezing tests immediately. The proportion chosen was 2 per cent, because it was thought that this amount would not cause the cement to set before it could be placed in the freezing plant, which was some distance from where the briquettes were made. In this second series of tests, Owl Portland, Dyckerhoff Portland, Star Stetton Portland, and AA Chicago Portland cements were used.

In the second attempt with Owl cement, the mortar with no soda required $2^h\,2^m$ for initial set. Using 50 per cent of soda, 9^m and 14^m were required, respectively, for initial and hard

set, 35^m were required for initial set when 10 per cent of soda was used, and 45^m when 5 per cent was used.

Dyckerhoff cement required 30 per cent or 15 c.c. of water to make a plastic mortar, and required $3^h\ 13^m$ to attain an initial set when no soda was used. Using 4 %, 2 %, and 1 % of soda, the initial set was obtained respectively in 20^m, 19^m, and 40^m, and hard set in $1^h\ 42^m$, $2^h\ 25^m$, and about $4^h\ 17^m$. These tests were repeated using 17 c.c. of water instead of 15 c.c.; and for the same proportions of soda, 4 %, 2 %, and 1 %, the time of initial set was 44^m, $1^h\ 9^m$, and $1^h\ 51^m$ respectively.

Thirty per cent of water was required to make a plastic mortar with Star Stetton cement. This cement without soda, required $2^h\ 56^m$ to attain initial set: with 4 per cent of soda $1^h\ 27^m$: with 2 per cent $1^h\ 40^m$ in one case and 56^m in another; with 1 per cent 46^m in one case and $1^h\ 23^m$ in another. To set hard the cement with 4 per cent of soda required $2^h\ 20^m$, with 2 per cent of soda $2^h\ 23^m$, and $1^h\ 30^m$, and with 1 per cent of soda $1^h\ 54^m$. The irregularity in these tests seemed to be due to one mortar being slightly wetter than the other.

AA Chicago Portland Cement required 20 per cent of water to make a plastic mortar,

and took $2^{h}\ 00^{m}$ to attain initial set when no soda was used. Using 4 o/o, 2 o/o, and 1 o/o of soda initial set was attained in 10^{m}, $1^{h}\ 2^{m}$, and $1^{h}\ 38^{m}$ respectively. There was some irregularity in getting the time required for the cement to set hard, due to lack of time to wait properly.

Table 1 shows a summary of the results of mixing bicarbonate of soda with cement.

Table 1.

Effect of Bicarbonate of Soda on Activity of 4 Cements.

Bicarb. Soda o/o Cement.	Amount of Water o/o of Cement.					Time Required for Initial Set, Minutes.					Time Required for Hard Set Minutes.				
	Owl 1st trial.	Owl 2nd trial.	Dycher-hoff.	A A Chicago	Star Stetten.	Owl 1st trial.	Owl 2nd trial.	Dycher-hoff.	A A Chicago.	Star Stetten.	Owl 1st trial.	Owl 2nd trial.	Dycher-hoff.	A A Chicago.	Star Stetten.
50		24					9					14			
25	24					*					*				
12½	24					2					12				
10		24					35								
10		24					33								
10		30					†22					86			
8	24					4					21				
5		24					9					14			
5		24					45								
4	24		34	22	30	21		44	10		52		145	20	
4	24		30	22	30			20	9	87			102	125	140
2			30	22	30			19		56			85		90
2			34	22	30			69	62	100			‡344	‡159	173
1			30	26	30			40	98	46			‡257		§111
1			34	22	30			111	90	83					
0	24	24	30	20	30		39	193	120	176	105				

* Cement set before it could be worked.

† Note amount of water. ‡ Approximate.

§ This was a little drier than others in the series.

Effect of Plasticity. In considering the tests with Star Stetton cement it was stated that the irregularity was probably due to one mortar being more or less plastic than the other. To see if such conditions would vary the time of set, tests were made with a constant quantity, 50 grams, of AA Chicago Portland cement, a constant quantity, 4 per cent, of soda, and different quantaties of water. Using 8, 10, 11, and 12 c.c. of water, the time required for initial set was respectively 10^m, 12^m, 10^m, and 24^m. Using 14 c.c. of water no initial set was obtained in $1^h\ 35^m$. These results are shown in table 2.

Table 2.

Effect of Plasticity on Activity of Cement.

4 per cent of Bicarbonate of Soda and 50 grams of A.A Chicago Portland Cement were used.

Reference Number.	C.C. Water.	Time Required for Initial Set Min.
1	8	* 10
2	10	12
3	11	9
4	11	10
5	12	24
6	14	not in 95

* Too dry to make a good pat.

Conclusions as to effect of Soda on Activity of Cement. From the results of the above tests it is seen

that the time of set can be shortened in proportion to the amount of soda used; but no definite rule for the amount to be used can be deduced. The results obtained show, however, that the addition of 4 per cent of soda decreased the time required to attain initial set 50 to 90 per cent, or an average of 70 per cent; with 2 per cent of soda, the time was decreased 40 to 70 per cent, or an average of 55 per cent; and with 1 per cent of soda the time was decreased 20 to 60 per cent, or an average of 40 per cent. It is also seen that the time required to attain initial set is affected by the amount of water used. A general conclusion might be stated as follows: The time required for cement mortar mixed with bicarbonate of soda to attain initial set decreases as the amount of water decreases, and as the amount of soda increases.

Tests with Potash Lye and Salt. To determine the the proportions of potash lye and salt mixture to use, the same method was employed as in determining the proportions of soda. Owl Portland cement was used, the same as in the first soda tests, and it required $1^h 45^m$ to set hard. Adding $\frac{1}{2}$ %, 1 %, 2 %, and 6 % of the potash lye and salt mixture, initial set was attained in each case in 8 minutes; and with $12\frac{1}{2}$ % and 16 % the

initial set was attained in 10^{m} and 39^{m} respectively. Using 25 % and 50 % of the mixture no initial set was attained in $1^{h}\,57^{m}$ and $1^{h}\,48^{m}$ respectively. With 1 %, 2 %, 6 %, and $12\frac{1}{2}$ % of the mixture, hard set was attained in 24^{m}, 34^{m}, 48^{m} and 59^{m}.

The results in Table 3 show that this mixture of potash lye and salt causes the time of set to increase as the per cent of the mixture increases.

Table 3.

Effect of the Potash Lye and Salt Mixture on Activity of Cement.

24 grms of Owl Cement and 24 per cent of Water.

Amount of Potash Lye and Salt Mixture per cent of Cement.	Time Required for	
	Initial Set Min.	Hard Set Min.
0	—	105
$\frac{1}{2}$	8	—
1	8	24
2	9	34
6	8	48
$12\frac{1}{2}$	10	59
16	39	—
25	not in 117	—
50	" " 108	—

There was the same reason for doubting these results as for doubting the results of the first activity tests with cement and soda, because

the same brand of cement was used in both cases; and therefore it was thought well to make another series of activity tests with the potash lye and salt mixture. It was necessary, however, to start the freezing tests before the second series of activity tests could be made, and it was decided to use 4 per cent of the mixture, because it was thought from a study of the preceding experiments that this amount would not cause the cement to set before it could be placed in the freezing plant. But before the second series of activity tests could be made, it was proved conclusively by the freezing tests, that the potash lye and salt mixture was very injurious to the cement; and therefore it was decided to abandon all the tests with this mixture.

Freezing Tests.

As mentioned in the introduction, the intention of this thesis is to investigate the effect of freezing cement mortar. It is generally conceded that cement mortar is injured when frozen and thawed alternately, and not injured when it is frozen continuously. With this fact in mind it was decided to make 7-day and 28-day tests under three different conditions; namely, (1) allow the cement to set in

water at the ordinary working temperature, (2) freezing the cement mortar continuously, and (3) freezing and thawing it alternately. For each of these conditions briquettes composed of 1 part cement and 2 parts sand were made in four ways as follows: (1) mixed with fresh water; (2) mixed with brine containing 37 per cent of salt, the proportions for a temperature of -5° F., the temperature to which the briquettes were to be exposed; (3) mixed with fresh, bicarbonate of soda equal to 2 per cent of the weight of the sand and cement having been added to the dry mixture; and (4) mixed with fresh water, 4 per cent of the dry mixture of potash lye and salt of proportions heretofore described having been added to the sand and cement. This plan reqiured the making of 144 briquettes for one series of tests.

Arrangements were made to use the refrigerating plant of the Agricultural College. This plant is an ammonia-evaporating system, the brine being cooled to an average temperature of -5° F. According to the rule mentioned earlier in this paper, if cement is mixed with a salt solution and is to be subjected to a temperature of -5° F. the solution should contain 37

per cent of salt.

One set of 144 briquettes were made in an afternoon. Of these one third were left in the cement laboratory to set in water in the ordinary way to serve as a standard for comparison: and of the remaining two thirds one half was to frozen and thawed alternately, and the other half was to be frozen continuously. Half of the briquettes in each of these groups were to be broken when 7 days old and half when 28 days old, but because of lack of room to carry on more than one set of experiments at one time all the briquettes were broken when 7 days old. As soon as the briquettes were made, those that to be frozen were hurried to the refrigerating room where the temperature is alway below 32° F. The briquettes were left in the moulds and kept in this room for 24 hours. Then those that were to be frozen steadily and also those that were to be frozen and thawed alternately were put into separate cans. The test pieces were then surrounded with water and frozen by setting the cans into the brine of the freezing tank. After twenty-four hours the briquettes to be frozen and thawed alternately were placed in a cupboard whose temperature

ranged from 80° to 90° F. and left there for 24 hours. This process was repeated each 24 hours for seven days, care being taken to have the briquettes thoroughly thawed at the end of the seventh day. The briquettes that were being frozen continuously were put into the same hot cupboard when they were six days old and allowed to thaw for 24 hours.

Seven days after the briquettes were made, they were subjected to the standard tensile test. A record of this test affords a means for determining whether or not using soda, or brine, or potash lye and salt, had prevented damage by freezing, and whether or not the materials were injurious to the cement.

Table 4 shows the results of these tests. It will be seen that they were not very satisfactory as so many briquettes were lost in storage by the pressure of briquettes piled on top. The results do show, however, that the brine, the bicarbonate of soda, and also the potash lye and salt mixture are injurious to the cement. This is particularly noticeable with the potash lye mixture, as all of the briquettes containing this material were too tender to be tested with the machine but were easily

pulled in two with the fingers.

Table 4.

Tensile Strength, in pounds per sq. in., of Briquetts of 1-2 Mortar 7 Days Old.

Material Used to Prevent Damage by Freezing.	Set in Water at about 65°F.	Alternate Freeze and Thaw.	Continuous Freeze.
Nothing.	Av. of 10 = 148	Broke in handling.	
Bicarbonate of Soda	Av. of 9 = 11.9	Av. of 6 = 51	Av. of 5 = 75
Potash Lye and Salt.	All broke by pulling with fingers.		
Satt Brine	Av. of 9 = 88	Broke in handling	

The results in table 4 are very unsatisfactory. It was thought that this was due to the fact that the briquettes were frozen in water, as when taken from the water they seemed to be thoroughly saturated and looked soggy. The briquettes were frozen in water because it was belived that this treatment was a close parallel to the ordinary method of permitting briquettes te set in water. There is a possibility that the character of the results was due to the fact that all the briquettes were not well made. It was a laborious task to make 144 briquettes in an afternoon, and therefore some of the last may not have been thoroughly compacted in the molds as the operator may have been fatigued

However an attempt was made in dividing the briquettes to eliminate this source of error. It was decided to make another series of tests leaving out the 28-day tests and freezing the briquettes without surrounding them with water.

In this second series twelve of each kind of briquettes were put into the refrigerator room whose temperature was 28°F. and kept in the molds for 24 hours. Six of each kind of briquettes were put into one pail then and the other six into another pail. The pails were then set into the brine the temperature of which at the time was -10°F. After 19 hours one pail was taken from the brine which was then at 0° F, and put into a cupboard whose temperature was 80° F. In 24 hours these briquettes were put back to freeze, and were frozen for 24 hours. They were then thawed and frozen three more times. Before breaking them all of these briquettes were kept for seven hours in a room whose temperature was 85° F.

Table 5 shows the results of these tests. These results show that the cement was injured by the brine, by the soda, and also by the potash lye and salt mixture as before.

Table 5.

Tensile Strength, in pounds per sq. in., of Briquettes of 1-2 Mortar 7 Days Old.

Material Used to prevent Damage by Freezing.	Set in Water at about 65°F.	Alternate Freeze and Thaw.	Continuous Freeze.
Nothing.	Av. of 6 = 236.	Av. of 6 = 172.	Av. 6 = 40.
Bicarbonate of Soda.	Av. of 5 = 124.	Av. of 5 = 63.	Av 6 = 56.
Potash Lye and Salt.	Av. of 6 = 72.	Broke by pulling with fingers.	Av. of 3 = 30 others broke with fingers.
Salt Brine.	Av. of 6 = 157.	Av. of 6 = 141.	Broke in handling.

Conclusions.

Effect of Salt. The results of the experiments show that salt is injurious to cement and decreases its strength. Mr Sabin, U.S. engineer, says in his book on Cement and Concrete, page [illegible] concerning some extensive experiments he had made to determine the effect of salt on the strength of cement: "The results indicate that Portland mortar made in low temperatures, to be immersed in cold water are improved by fifteen to twenty per cent of salt in the gaging water, but that more than five per cent of salt is deleterious for mortars exposed to the air only". As 37 per cent of salt was used in the gaging water employed in the tests of this thesis it is reasonable to say that the large amount of salt used accounts for

the low strength of the mortar containing salt, and justifies the writer's conclusion stated above.

Effect of Soda. The preliminary tests to determine the effect of bicarbonate of soda on the activity of cement show that the cement's activity was increased very materially. The rate of increase is directly proportional to the amount of bicarbonate of soda used.

In connection with the same tests efflorescense was very noticeable. The pats of mortar containing soda, after standing a short time, became coated with a white substance undoubtedly caused by the soda as the mortar when mixed without it was not affected.

The freezing tests show that the increasing action of the soda was not helpful in preventing damage to the cement by freezing, but show that the soda is injurious to the strength of the mortar. It may be that the damage done was due to the soda and not to the freezing, but this can not be proved.

As far as the object of the thesis is concerned, the use of soda fails to be justified because of its deleterious effect on the cement mortar. However it is very likely that soda could be used with cement mortar where

the latter is to be used as a coating and it is desired to have it impermeable and set quickly, as for example in stopping a leak.

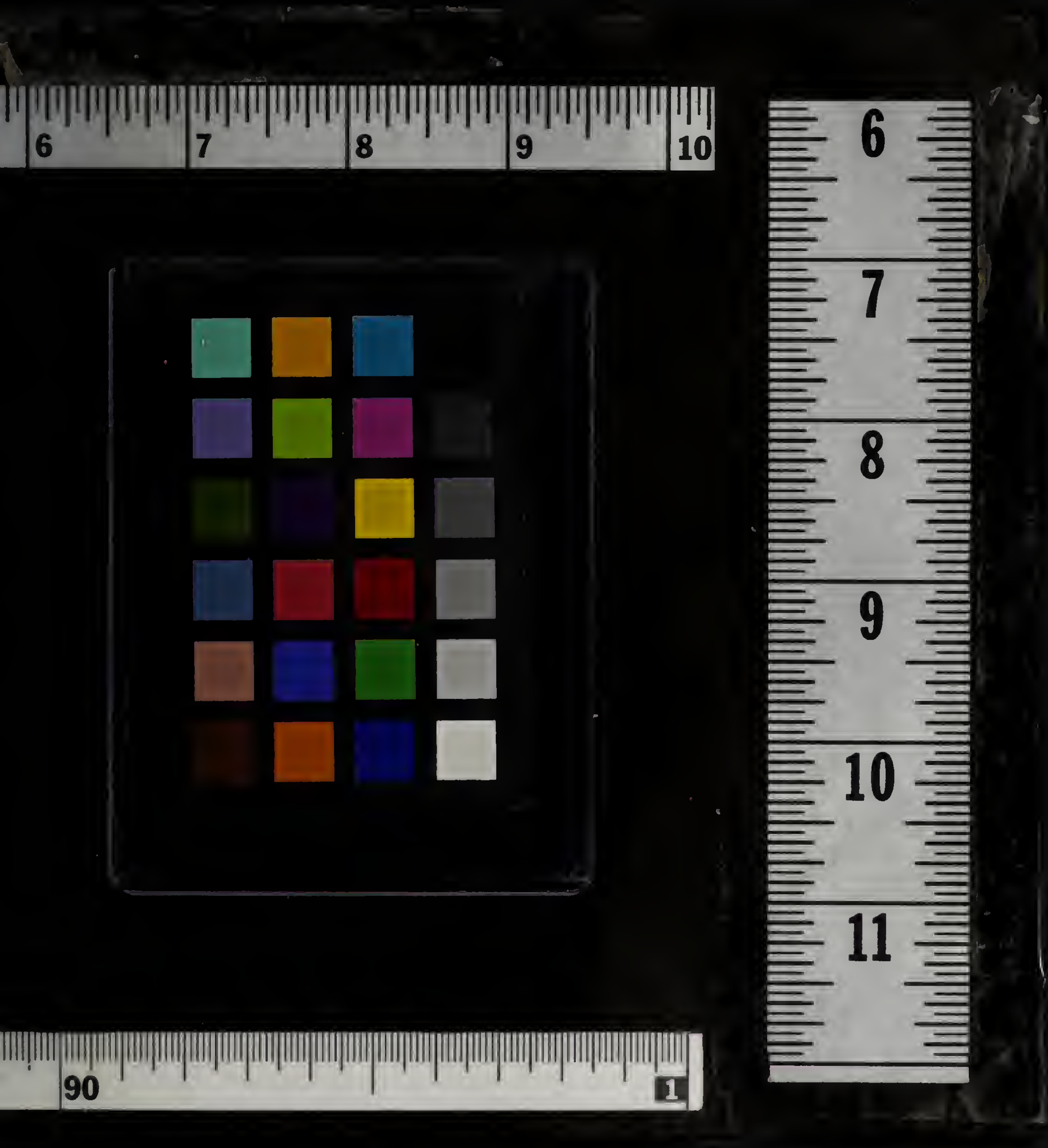
Potash Lye and Salt Mixture. There is no doubt that the potash lye and salt mixture is very injurious to cement mortars. The only justification for its use that can be suggested is the same as for the soda, namely where it is desired to have an impermeable and quick setting mortar.

Freezing. The results of the tests contradict the general opinion that continuous freezing is less detrimental to cement mortar than alternate freezing and thawing. It is difficult to explain these anomolous results. However it may be that the soda used in the cement, and the fact that the briquettes were thawed in a room whose temperature was very high, is the cause of this contradiction. Being put into such a warm place after having been frozen, it is possible that the briquettes became thoroughly thawed very quickly and that the mortar then had an opportunity to partially set before it was frozen again. This is purely an assumption and should be tested farther before such a statement could be made with any degree of assurance. In making such tests

the following plan might be followed. Make 36 briquettes of mortar containing soda and 36 briquettes of mortar without soda. Of each set subject 12 to the ordinary test breaking 6 at 7 days and 6 at 14 days: alternately freeze and thaw 12 for 7 days breaking 6 at 7 days and putting the others in a warm place, and then break them at 14 days; the remaining 12 should be frozen steadily for 7 days, when 6 should be broken and the remaining 6 put into a warm place until they are 14 days old and then broken. By doing this it could be proved whether or not the bicarbonate of had caused the cement subjected to alternate freezing and thawing to set while it was thawed and also if the cement without the soda did not have a chance to set while it was thawed. If the briquettes that had been alternately frozen and thawed did not test as high at the end of 14 days as the ones continually frozen do, it will be safe to say that the sod and the quick thawing cause the alternately frozen and thawed to test higher at the end of the seven days.

CONCLUSION. It is evident that enough tests were not made in this work to enable

any definite conclusions to be drawn. This was due to lack of time and room in which to work properly. However the writer hopes that his work has not been in vain, and that his results will prompt some one to investigate this subject more thoroughly, and he believes that further research may discover results of value to the engineering profession.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